Enhanced supercapacitor performance of Ni intercalated V_2O_5 composite via facile solvothermal synthesis

Dissertation submitted to

Visvesvaraya National Institute of Technology, Nagpur

In partial fulfilment of the requirements for the award of

the degree

Master of Science

in

Chemistry

By

Praveen Kumar Lamba (MS23CHM019)

Under the Guidance of

Dr. Ramani V. Motghare

Associate Professor

Department of Chemistry

Visvesvaraya National Institute of Technology

Nagpur, 440010 (India)

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Declaration

I, Praveen Kumar Lamba hereby declare that this dissertation titled **"Enhanced supercapacitor performance of Ni intercalated V_2O_5 composite via facile solvothermal synthesis"** was carried out by me in the Department of Chemistry of Visvesvaraya National Institute of Technology, Nagpur. The work is original and has not been submitted earlier, whole or in part, for the award of any degree/diploma at this or any other Institution / University**.**

Praveen Kumar Lamba

(MS23CHM019)

Date:

Certificate

This is to certify that the dissertation titled **"Enhanced supercapacitor performance of Ni intercalated V_2O_5 composite via facile solvothermal synthesis"** submitted by **Praveen Kumar Lamba** in partial fulfilment of the requirements for the award of the degree of **Master of Science in Chemistry, VNIT Nagpur.** The work is comprehensive, complete, and fit for final evaluation

Dr. Ramani V. Motghare

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VISVESVARAYA NATIONAL INSTITUTE OF TECHNOLOGY, NAGPUR

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Acknowledgement

I would like to express my gratitude to everyone who has contributed to the successful completion of this project. First and foremost, I am extremely grateful to my supervisor, **Dr. (Mrs.) Ramani V. Motghare**, whose expertise, guidance and support have been invaluable and unparalleled throughout this tenure.

I would also like to express my gratitude to the Head of the Department, **Dr. Sujit K. Ghosh**, for his commitment towards science and research and for providing all the necessary facilities available in the department. My sincere thanks to **Dr. Anupama Kumar**, the former HoD, who has helped and motivated me in the journey of post-graduation.

Through this, I would acknowledge the contribution of the Electroanalytical Laboratory, Dept of Chemistry, VNIT Nagpur, towards the successful completion of this dissertation. A special thanks to the PhD scholars, Gaurav Deolikar, Dr. Prashu Jain, Anuj Shrivastava, Shobha Kawad, and Sanskruti Kanfade who have not only helped me throughout this journey with technical assistance but also nurtured me with their mental and moral support.

I would like to appreciate the constructive criticisms and sharing of knowledge by my batch mates that helped me complete this project. Thank you for supporting me through the thick and thin.

I would like to express my heartfelt thankfulness to my family for their nonstop support, belief and blessing.

Abstract

Vanadium pentoxide (V_2O_5) has emerged as a highly promising electrode material for energy storage applications, owing to its unique layered crystal structure facilitates efficient ion intercalation and de-intercalation. This structural feature, combined with its high theoretical capacitance, makes V_2O_5 an attractive candidate for supercapacitors. However, its lesser electrical conductivity and limited cycling stability hinder practical supercapacitor performance. To address these challenges, we have successfully synthesized a series of Ni-intercalated V_2O_5 composites with varying Ni content (10%, 20%, 30%, and 40%) via a facile solvothermal method. The incorporation of Ni, a highly conductive transition metal, is aimed at enhancing the electrochemical performance of V_2O_5 by improving conductivity and facilitating faster charge transport. The structural and thermal properties of the synthesized composites were systematically studied using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), Field emission scanning electron microscopy (FE-SEM), and thermogravimetric analysis (TGA). The XRD patterns confirmed the successful formation of V_2O_5 and the effective incorporation of Ni without the formation of secondary phases. FE-SEM images revealed a well-defined nanostructure with uniform dispersion of Ni within the V_2O_5 matrix, while FTIR spectra supported the interaction between V_2O_5 and Ni. TGA analysis demonstrated the enhanced thermal stability of the Ni-intercalated composites compared to pristine V_2O_5. In a three-electrode setup, electrochemical performance was evaluated using cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) techniques. Among the series, the composite containing 20% Ni intercalated V_2O_5 exhibited the highest specific capacitance of **1500 F/g**, along with excellent energy and power densities, highlighting the synergistic effect of Ni intercalation. The superior performance can be attributed to improved electrical conductivity, enhanced surface area, and faster ion diffusion pathways introduced by the Ni nanoparticles. These results suggest that Ni-intercalated V_2O_5 composites are promising candidates for high-performance supercapacitor electrodes.

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Chapter 1
GENERAL INTRODUCTION

1. Introduction

The global demand for energy and power has risen significantly in recent decades. While numerous energy sources are available on Earth, including both renewable and non-renewable options, these are not ideal for long-term use [1]. As the name suggests, non-renewable energy sources are finite and will be depleted over time. On the other hand, renewable energy sources such as solar, wind, and tidal energy, while sustainable, have limited availability. Considering these factors, the escalating need for energy has driven the development of energy storage technologies. These portable storage systems have made considerable strides in electrochemical energy storage applications, including batteries and supercapacitors. Key performance metrics such as energy density (Wh/kg) and power density (W/kg) are often evaluated in these systems [2-4].

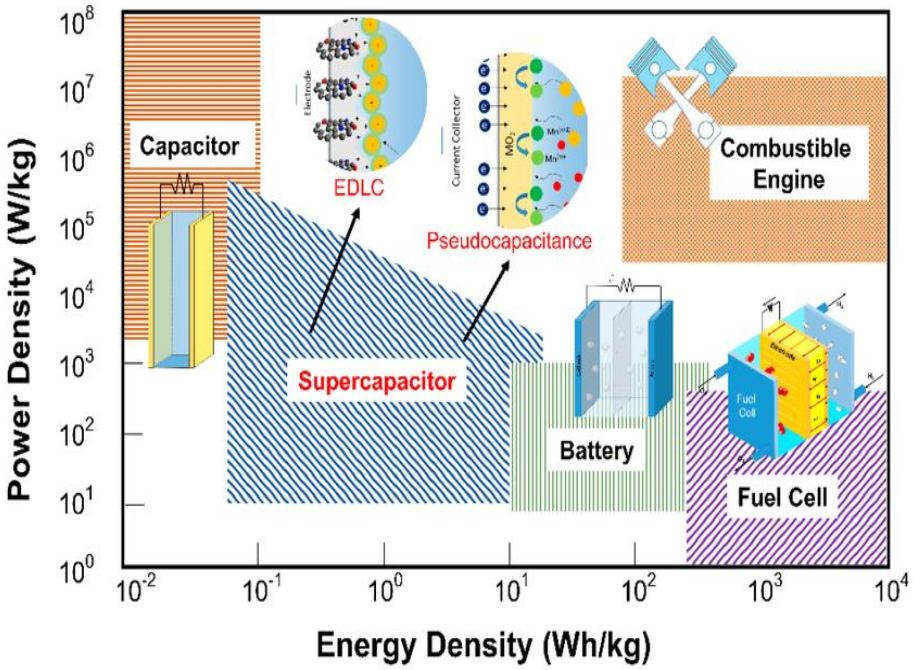

Fig.1.1 Ragone plot.

The Ragone plot, in **Figure 1.1**, delineates the correlation between specific power and specific energy across a range of energy storage technologies, including fuel cells, electrochemical batteries, and electrochemical double-layer capacitors (supercapacitors) [5].

It is evident from the plot that fuel cells and battery-based systems typically exhibit energy densities, implying substantial energy retention capabilities. However, these systems are characterized by limited power densities, indicating relatively sluggish charge-discharge kinetics. In contrast, conventional capacitors demonstrate exceptional power densities, enabling rapid energy delivery and uptake, albeit at the expense of diminished energy densities, thereby constraining their total energy storage capacity [6].

To overcome these inherent trade-offs, supercapacitors have been introduced as a hybrid energy storage solution. As observed in the Ragone plot, supercapacitors offer a favorable balance between specific energy and specific power [7]. This indicates that supercapacitors are capable of delivering moderately high energy densities while maintaining superior charge-discharge rates, rendering them highly suitable for applications that necessitate both high power output and efficient energy buffering [8].

1.1 Supercapacitors

Supercapacitors, also referred to as ultracapacitors, are energy storage devices that utilize electrostatic fields to store electrical energy, in contrast to traditional batteries that rely on chemical reactions. These devices exhibit high power density, enabling rapid charge and discharge cycles, and possess an exceptional cycle life, which makes them well-suited for applications requiring fast energy delivery. Their ability to provide quick bursts of power makes them ideal for uses such as regenerative braking systems in electric vehicles, where they capture energy during braking and release it quickly for acceleration. Supercapacitors are also increasingly used in energy storage systems for renewable energy integration, where rapid cycling capabilities are beneficial for stabilizing power supply fluctuations [3].

However, supercapacitors have a relatively low energy density compared to conventional batteries, which limits their ability to store large amounts of energy over extended periods. As a result, they are often integrated with batteries in hybrid energy storage systems to optimize the overall performance, combining the high-power density of supercapacitors with the high energy density of batteries. Recent advances in materials, such as graphene

and carbon nanotubes, have led to significant improvement in supercapacitors' performance, enhancing both energy storage capacity and cost effectiveness. These innovations continue to expand the potential applications of supercapacitors, ranging from consumer electronics to grid-level energy storage, and offer promising solutions to address global energy storage challenges [8].

1.2 Classification of supercapacitors

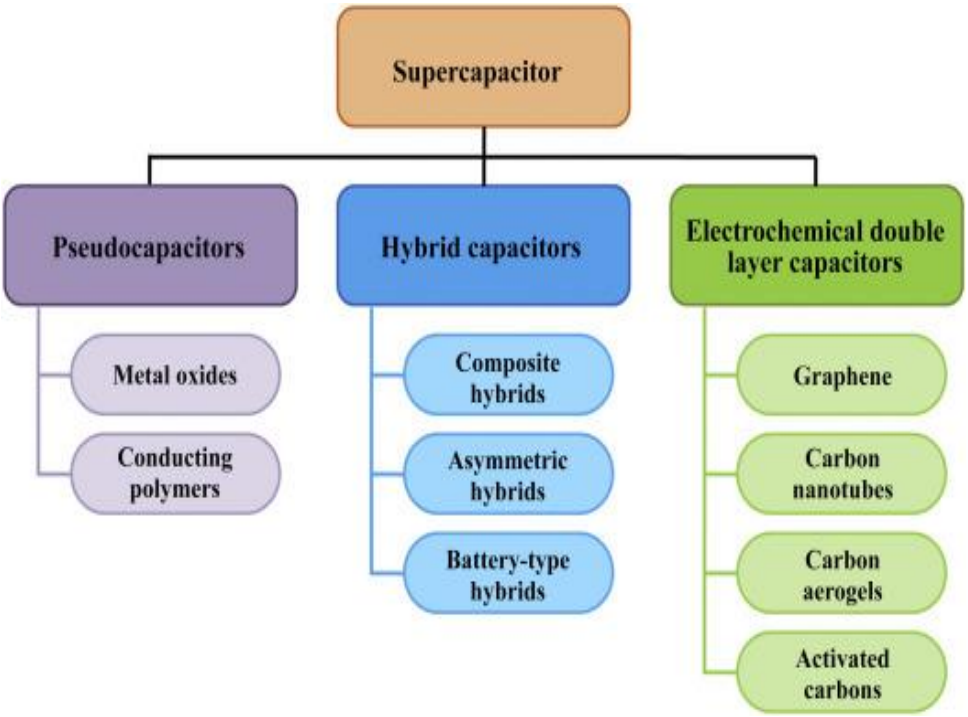

Fig.1.2 Classification of supercapacitors.

Supercapacitors are generally divided into **electric double-layer capacitors (EDLCs), pseudo-capacitors**, and **hybrid supercapacitors**. EDLCs store energy using two parallel electrodes separated by a thin, selectively permeable layer and placed in a special liquid called an electrolyte. When voltage is applied, charges build up on the surfaces of the electrodes at the point where they meet the electrolyte, forming what's known as a Helmholtz double layer due to polarization [9]. The process involves ions from the electrolyte attaching to the electrode surfaces, and when charging or discharging, ions and

electrons move back and forth, forming two charged layers separated by a layer of solvent molecules. This energy storage is pure physical, relying on electrostatic forces.

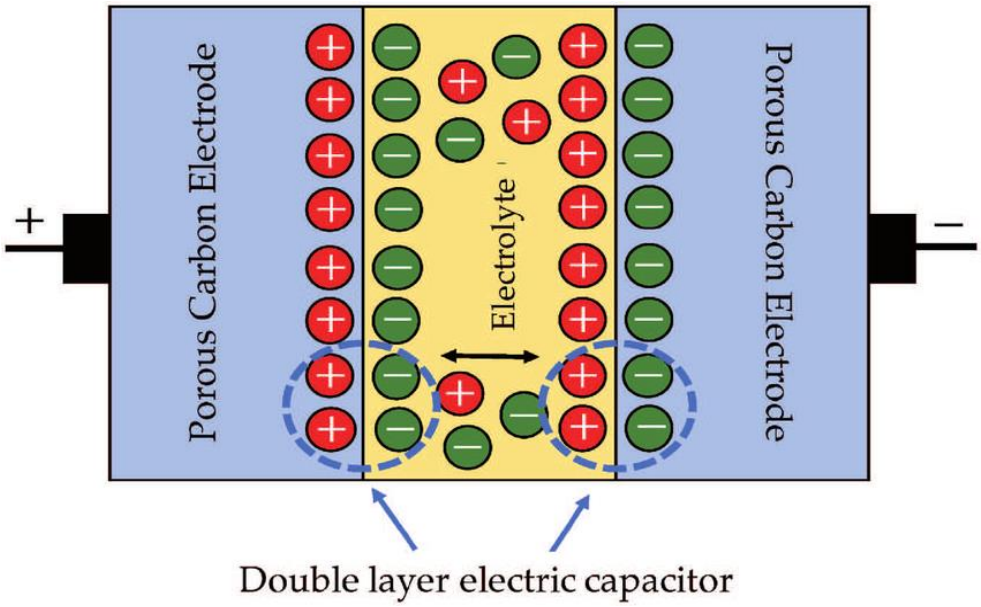

Fig.1.3 Electric Double layer capacitor.

On the other hand, pseudo-capacitors store energy through quick chemical reactions known as fast redox (reduction-oxidation) reactions. When voltage is applied, electrons are transferred, which changes the chemical state, like the oxidation state of the electrode material [10].

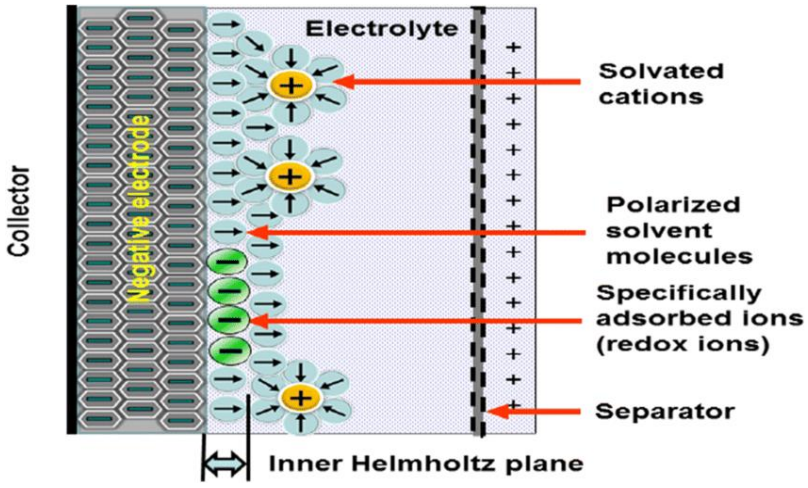

Fig.1.4 Pseudo-capacitors.

Lastly, hybrid supercapacitors combine features of both EDLCs and pseudo-capacitors. In these, one electrode stores energy through redox reaction (like in pseudo capacitors), while the other stores it using separation without chemical change (like in EDLCs), offering a balance of high energy and fast charge/discharge capability [11].

1.3 Components of supercapacitors

A supercapacitor mainly has two important components: the electrodes and the electrolyte. The electrolyte is a liquid solution, either water-based or organic, that helps ions to move when the supercapacitors charge or discharge. The electrodes are the materials placed inside the electrolyte and play a key role in storing and moving electrical charge. These electrodes have a large surface area, which helps them store more energy. How well a supercapacitor works mostly depends on the type of materials used for the electrodes**.**

1.3.1 Electrode materials

The supercapacitor electrode is mainly made up of four types of materials:

1) **Carbon-based materials**: These are commonly used in EDLCs. They have a large surface, adjustable pore sizes, and good electrical conductivity. While they provide high power density, their energy density is relatively low. Some common examples include activated carbon, carbon dots, carbon nanotubes (single and multi-walled), fullerenes, and graphene. Most commercial supercapacitors use nanoscale carbon materials [12,13]. These materials, especially those with sp^2 hybridized carbon, offer strength, porosity, and enhanced electrical performance due to the presence of free-moving electrons.

2) **Conducting polymer:** materials like polyaniline (Pani) and polypyrrole (PPy) are often used in energy storage because of their good electrical conductivity. They also perform well in supercapacitor applications. However, their chemical and mechanical stability can be a concern over time, which limits their long-term use 14,15].

3) **Transition metal oxides (TMOs):** These materials offer high specific capacitance (between 100-2000 F/g) and better storage performance than carbon or polymer-based electrodes. Ruthenium oxide (RuO_2) is one of the most well-known TMOs used in supercapacitors. TMOs are popular because metals are widely available and easy to work with. They also have excellent electrical properties, making them suitable for various energy-related applications [16].

4) **Metal organic framework (MOFs):** MOFs are a new class of materials that combine metal ions or clusters with organic molecules to form a highly porous structure. These materials have shown great potential in supercapacitors due to their extremely high surface area and tunable properties, making them capable of storing large amounts of energy. MOFs can also be designed them promising candidates for next-generation energy storage devices [17,18].

1.3.2 Electrolyte

Electrolytes are an essential part of supercapacitors because they allow ions (charged particles) to move between the electrodes during charging and discharging. The electrolyte is made by dissolving certain chemicals (called solutes) into a liquid (called a solvent), and this mixture breaks into positive and negative ions. These ions are what carry the electric charge inside the supercapacitor. The performance of a supercapacitor depends heavily on the type and concentration of the electrolyte [19]. A higher concentration of ions usually means better electrical conductivity. However, the electrolyte must also be stable and should not react with the electrode materials. If there is a chemical reaction between them, it can reduce the life and performance of the device. Another important factor is the viscosity of the electrolyte. If the liquid is too thick, it becomes difficult for ions to move quickly. Low viscosity electrolytes are preferred because they allow faster ion movement, which improves the charge/discharge rate of the supercapacitor. There are different types of electrolytes used in supercapacitors:

1) **Liquid electrolyte:** These are most common type and usually involves water or organic solvents. Water is known as a universal solvent and works well with acids, bases, and salts. It offers high conductivity, especially when mixed with strong acids (like sulfuric acid) or alkalis (like potassium hydroxide). However, water-based electrolytes can only operate within a narrow voltage range (usually below 1.23V), which limits their energy storage capacity. They are also sensitive to temperature changes. For this reason, aqueous electrolytes are mainly used in applications where high power and fast charge/discharge are needed, but not high energy storage.
2) **Organic electrolytes**: These use organic solvents like acetonitrile or propylene carbonate. They offer a wider voltage window (up to 2.5- 3V), allowing the supercapacitor to store more energy. However, they usually have lower conductivity than aqueous electrolytes, are more expensive, and can be flammable or toxic.
3) **Polymer gel electrolytes:** These are a mixture of liquid electrolytes trapped inside a polymer matrix. They combine the benefits of solid and liquid electrolytes. Polymer gels prevent leakage and evaporation and are safer to use. They are also useful for creating flexible and wearable supercapacitors, which are gaining popularity in modern electronics. The importance of flexible supercapacitors is designed to bend, stretch, or twist, which makes them perfect for use in wearable electronics, foldable medical devices, and smart textiles. They are lightweight, easy to handle, and can operate in a wide range of temperatures. With polymer gel electrolytes, it is now possible to create energy storage devices that not only perform well but also integrate seamlessly into modern, flexible electronic systems [19,20].

Flexible supercapacitors have numerous advantages, such as ease in handling, being lighter in weight than conventional capacitors, reliability is higher, and provide operation temperature in a wider range.

1.4 Aim/Objectives of the experiment:

Synthesis of electrode materials effective for energy storage applications, mainly supercapacitors, followed by their characterization and electrochemical studies.

The main aim of this dissertation study is as follows:

- Synthesis of Nickel intercalated vanadium pentoxide via the solvothermal method.
- Characterization of synthesized metal oxide via techniques like as powder X-Ray diffraction (p-XRD), Fourier transform infra-red spectroscopy (FTIR), Field emission scanning electron microscopy (FE-SEM), Energy dispersive X-ray spectroscopy (EDX).
- Electrochemical studies of the synthesized materials, such as cyclic voltammetry (CV), and Galvanostatic charge-discharge (GCD).

Chapter 2

Literature survey

2. Literature survey

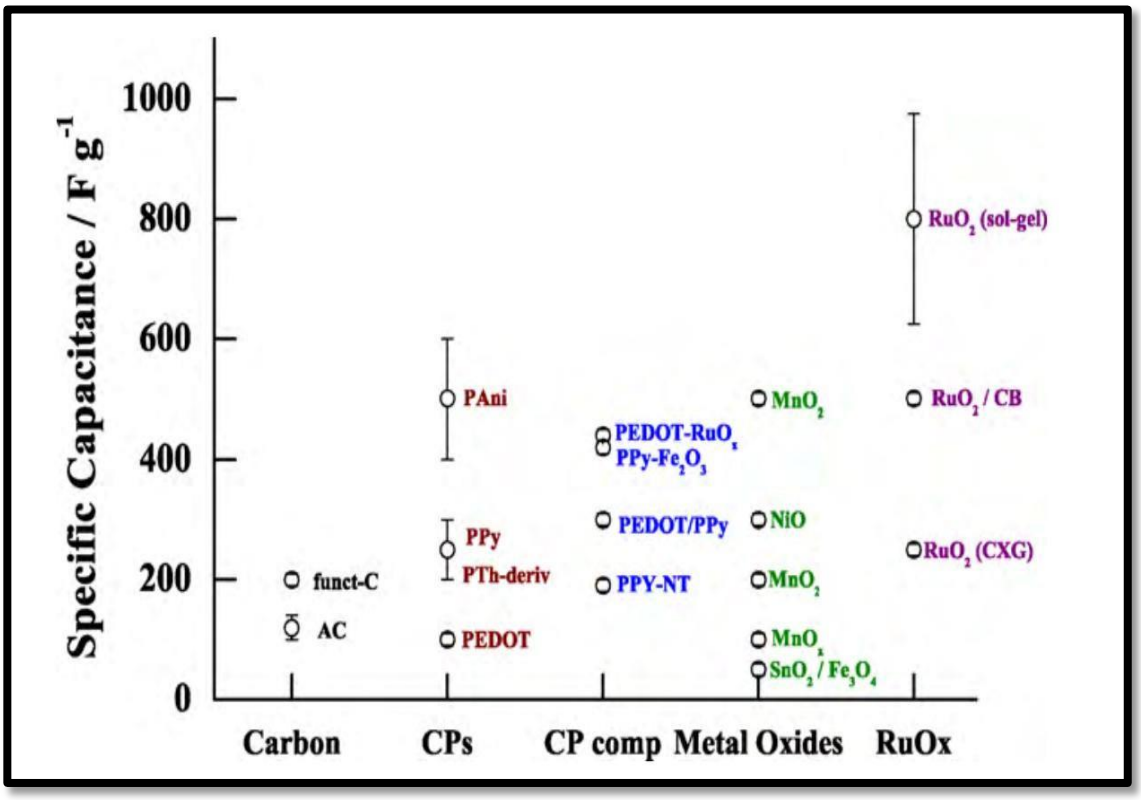

Fig. 2.1 A relative study of the specific capacitance of various electrode materials.

The given **Figure 2.1** shows how well different electrode materials perform in terms of specific capacitance (C_{sp}) when used in supercapacitors.

Among conducting polymers, polyaniline (Pani) performs very well, with a specific capacitance of about 700 F/g and very little loss in performance, even after thousands of charge-discharge cycles [21]. Another material, PEDOT (Poly(3,4-ethylenedioxythiophene)), especially in its heavier form, has a C_{ps} of around 200 F/g [22]. polypyrrole (PPy) is also widely used and reaches about 650 F/g. Polythiophene (PT), on the other hand, offers up to 480 F/g, but its performance drops by about 9% after 2000 cycles [23].

Carbon materials like activated carbon, carbon nanotubes (CNTs), and graphene are widely used due to their high surface area and excellent electrical conductivity [24, 25]. However,

their energy storage primarily occurs through electrical double layer capacitance (EDLC), which limits their overall capacitance. Activated carbon remains the most commercially used material, offering specific capacitance values typically in the range of 100-300 F/g, depending on porosity and surface modification. Carbon nanotubes (CNTs), due to their 1D structure and good electrical properties, exhibit capacitance values between 150-300 F/g, often enhanced through surface functionalization or composite formation. Graphene and reduced graphene oxide have gained attention for their high theoretical capacitance (~550 F/g) and actual performance values generally ranging from 200 to 400 F/g, especially when incorporated into hybrid structures. Carbon aerogels and carbon nanofibers also contribute to the field with moderate to high capacitance values (100-250 F/g), particularly when doped or combined with pseudocapacitive material. Overall, advancements in carbon electrode design continue to push the performance limits of electrochemical double-layer capacitors (EDLCs), with hybrid and hierarchical architectures leading recent developments [26].

2.1 Transition metal oxide

Transition metal oxides like Ruthenium oxide (RuO_2), is one of the most effective due to its excellent conductivity and stability over many cycles. However, its high cost makes it difficult to use on a large scale [27]. Cobalt oxide (Co_3O_4) has a very high theoretical capacitance (up to 3500 F/g), but in real applications, particles tend to clump together, reducing surface area and overall performance [28]. MnO_2 is a cost-effective and environmentally friendly alternative [29]. Depending on the synthesis method and structure, it can deliver specific capacitance in the range of 200-300 F/g. Its limitations include relatively low electrical conductivity, which has been addressed through hybridization with conductive substrates such as graphene or CNTs.

NiO possesses a high theoretical capacitance (~2584 F/g) and good electrochemical stability [30, 31]. However, similar to MnO_2, its performance is hampered by poor intrinsic conductivity. Techniques such as nanostructure approaches, such as the fabrication of nanowires or nanosheets, have been widely used to enhance surface area and electrochemical activity. V_20_5 shows attractive pseudocapacitive characteristics with a layered structure that facilitates ion intercalation [32]. Nevertheless, it suffers from low

electrical conductivity and structural instability during cycling, which can be mitigated by forming a composite with conductive polymers or carbon-based materials.

Some metal oxides have oxygen vacancies, which means missing oxygen atoms in their structure. These vacancies can improve the materials' conductivity and make them more active in chemical reactions, helping ions move more easily during charging and discharging. Ceria (CeO_2) naturally forms these vacancies and is a good example of such a material [33].

2.2 Hybrid and composite materials

To overcome the drawbacks of individual TMOs, hybrid materials have been developed by combining TMOs with conductive matrices like Transition metal Nanoparticles and conductive carbon materials which are known for their high electrical conductivity[34, 35]. These composites benefit from the synergistic effect, enhancing both conductivity and structural stability. For example, MnO_2/Ni nanoparticles, NiO/CNT, and TM nanoparticles/TMOs composites have shown significantly improved and cyclic durability.

Chapter 3

Materials: Experimental and Characterization Techniques

This chapter mainly explores the synthesis of electrode material and its characterization using various techniques, including FTIR, XRD, FE-SEM, EDS, and TGA.

3.1 Chemicals

All chemicals used in this work were high-purity, analytical grade that did not require additional purification before use.

Table 3.1: List of chemicals

Chemicals/ Reagents	Formula	Company and Purity
Ammonium metavanadate	NH_4VO_3	Sisco Research Laboratory, Extra pure AR, > 99%
Oxalic acid	$(COOH)_2$	Alfa Aesar, AR, > 99%
Potassium Hydroxide pellets	KOH	Sigma Aldrich, AR, ≥ 85%
Ethanol, Absolute	C_2H_5OH	Fisher Scientific, Chemical, 99.9%
Nickel Nitrate hexahydrate	$Ni(NO_3)_2.6H_2O$	Merck, 99.9%
Acetone	CH_3COCH_3	Merck, India, 99.5%
Nickel-foam	Ni	Sigma Aldrich, AR, ≥ 99.9%
Sulphuric Acid	H_2SO_4	Merck, India, 98%
Hydrochloric Acid	HCl	Fisher Scientific, 36.45%

3.2 Characterization techniques

1) Fourier transform Infra-Red spectroscopy (FTIR):

FTIR is an important analytical method used to identify chemical substances by examining how they interact with infrared (IR) radiation, which has wavelengths between 4000 cm^{-1} and 400 cm^{-1}. The basic principle behind IR spectroscopy is the study of the vibrational motions of molecules, such as bending, stretching, and twisting. vibration occurs at specific frequencies that are unique to the structure of each molecule. These frequencies are related to the types of bonds and functional groups (specific arrangements of atoms) within the molecule.

When IR radiation hits a molecule, the energy is absorbed if the frequency of the incoming radiation matches the frequency of the molecule's vibrations. This absorption causes the molecule to move from a lower energy state to a higher one. Since energy in these vibrations is quantized, absorption only occurs at specific frequencies corresponding to the difference in vibrational energy levels.

The resulting IR spectrum displays the intensity of light that is either absorbed or transmitted across various frequencies. The peaks in the spectrum, whether sharp or broad, are characteristic of specific functional groups or chemical bonds in the molecule.

FTIR is a more advanced version of traditional IR spectroscopy. In FTIR, an interferometer is used to split a single IR beam into two beams. These beams interact with the sample and then recombine to create interference patterns. A Fourier transform, which is a mathematical operation, is applied to convert the data from a time-based signal to a frequency or wavenumber-based signal. This process produces the final IR spectrum.

FTIR has significant advantages over traditional IR spectroscopy. It is more efficient, offering better resolution, high accuracy, and greater sensitivity, making it a more powerful tool for identifying chemical substances.

In the thesis work, the functional groups and bonding characteristics of the prepared samples were analyzed using an FTIR spectrometer (IR-Affinity-1, Shimadzu).

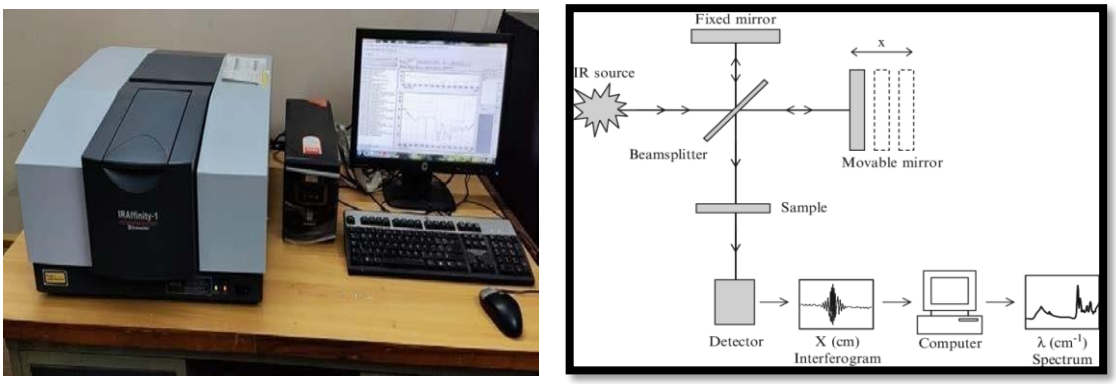

Fig. 3.1 FTIR instrument and Schematic diagram of the FTIR spectrometer.

2) Powder X-ray diffraction (PXRD):

PXRD is a technique used to study the structure and composition of materials. X-rays, usually produced by metal sources like copper (Cu), molybdenum (Mo), or silver (Ag), are created when fast-moving electrons hit the metal targets. These X-rays interact with the material being studied, whether it is crystalline or non-crystalline, and create diffraction patterns (shown in Fig 3.2). By analyzing these patterns, we can learn about important properties such as:

- Crystallinity
- Phase identification
- Size of the crystals
- Distance

The diffraction pattern depends on the arrangement of atoms in the material and follows a principle known as Bragg's law for crystalline substances:

$$n\lambda = 2dsin\theta \ldots\ldots\ldots\

This equation helps us understand how X-rays interact with the crystal and the diffraction pattern.

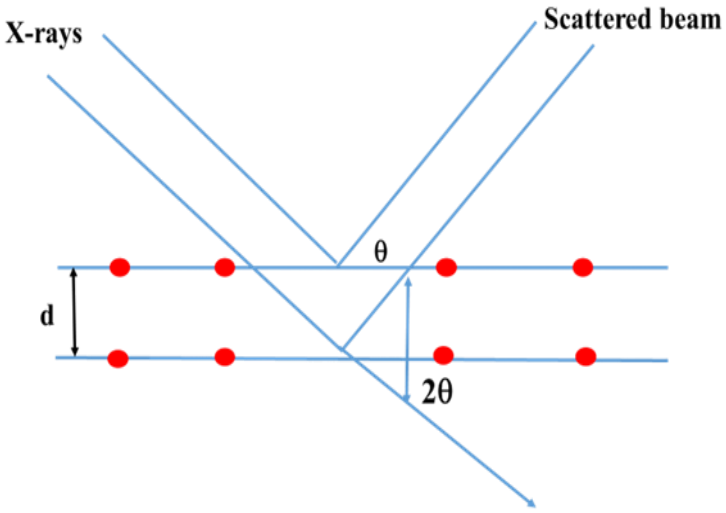

Fig. 3.2 X-ray diffraction pattern from crystal structure.

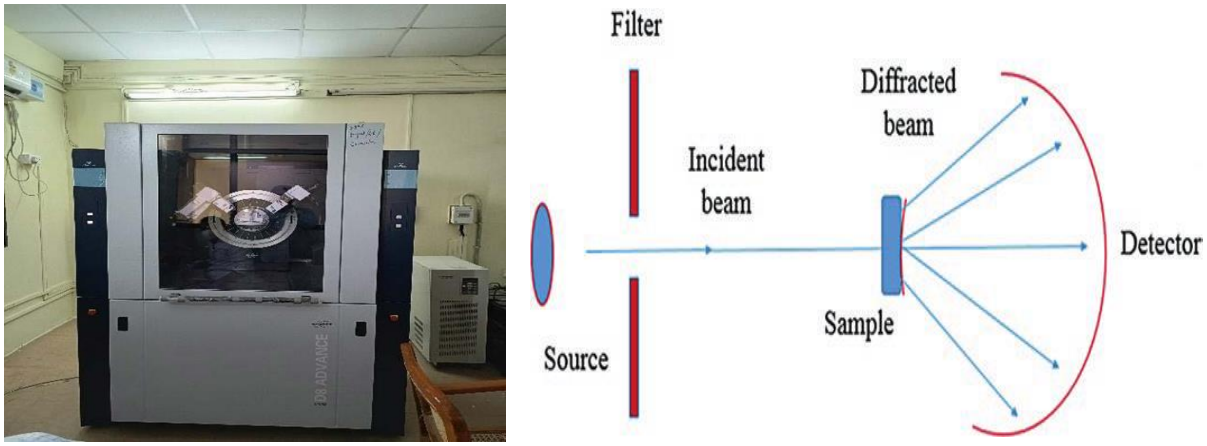

Fig. 3.3 P-XRD Instrument and its components.

The XRD data for the thesis were recorded using a Brucker D8 Advance Eco X-ray diffractometer from Karlsruhe, Germany.

3) Field Emission Scanning Electron Microscopy (FE-SEM)

Field Emission Scanning Electron Microscopy (FE-SEM) is one of the most advanced techniques for high-resolution imaging, widely used to analyse the surface morphology of materials. It is based on the principles of traditional Scanning Electron Microscopy (SEM),

where a high-energy electron beam (typically in the range of 1–30 keV) is focused onto the surface of a sample. This beam is directed using a system of electromagnetic lenses and deflectors.

Upon interaction with the sample, the primary electron beam generates various signals, including secondary electrons, backscattered electrons, and characteristic X-rays. These secondary electrons are collected and directed toward a detector using electromagnetic lenses, which ultimately produces high-resolution images of the sample's surface.

What distinguishes FE-SEM from conventional SEM is its field emission gun (FEG), which serves as the electron source. The FEG—often made from tungsten or lanthanum hexaboride—delivers an electron beam with significantly higher brightness and intensity, enabling extremely fine resolution and enhanced image quality.

FE-SEM provides detailed information on surface topography, composition, and structure at the nanoscale, along with an excellent depth of field, making it an indispensable tool for nanomaterial characterization.

The surface morphology of the samples prepared was imaged using a field emission scanning electron microscope (FESEM) JSM-6701F (JEOL, Tokyo, Japan)

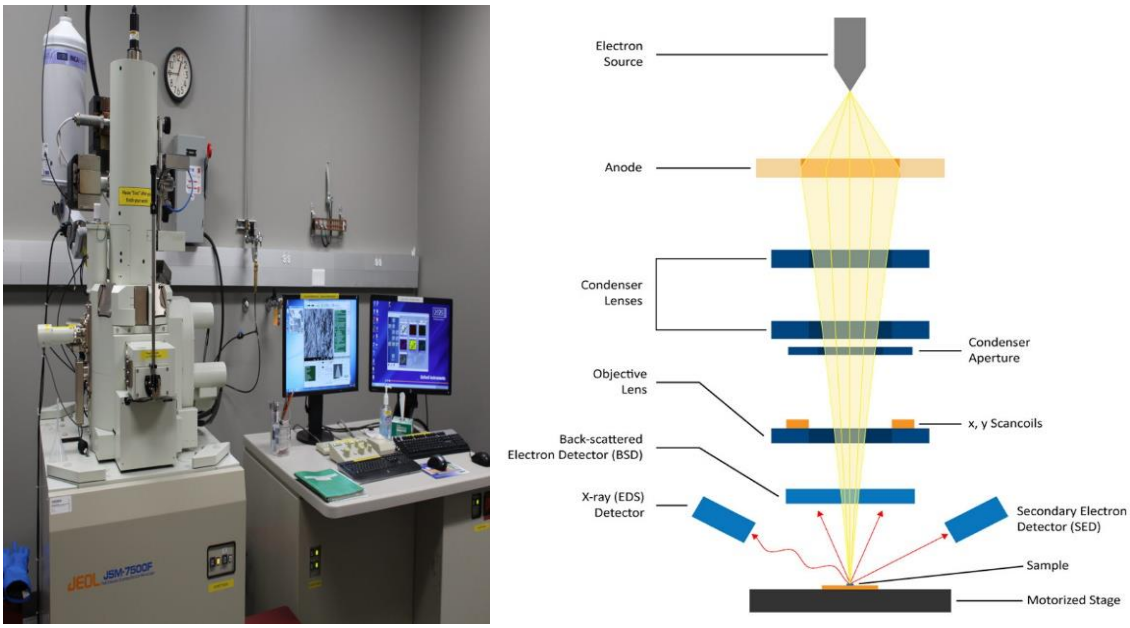

Fig. 3.4 SEM Instrument and its components.

4) Energy Dispersive X-ray Spectroscopy (EDS or EDX)

Energy Dispersive X-ray Spectroscopy (EDS) is a powerful analytical technique used for elemental analysis or chemical characterization of materials. It is commonly integrated with electron microscopy tools such as SEM or TEM.

In EDS, a focused high-energy electron beam interacts with the atoms in a sample. This interaction excites electrons in the inner shells of the atoms, ejecting them and creating vacancies. Electrons from higher energy levels then fall into these lower energy states, releasing the excess energy in the form of characteristic X-rays.

Each element emits X-rays with unique energy signatures, allowing for the identification and quantification of the elements present in the sample. Thus, EDS complements imaging techniques like SEM and FE-SEM by providing precise elemental composition data, often used to analyse inclusions, contaminations, or compositional distributions within materials.

The EDX Spectra of synthesized materials was recorded using a Joel apparatus [Model- JSM 5400] equipped with (FESEM) JSM-6701F (JEOL, Tokyo, Japan).

3.3 Synthesis of Materials

3.3.1 Synthesis of Transition Metal Oxides

Hydrothermal synthesis of vanadium pentoxide(V_2O_5)

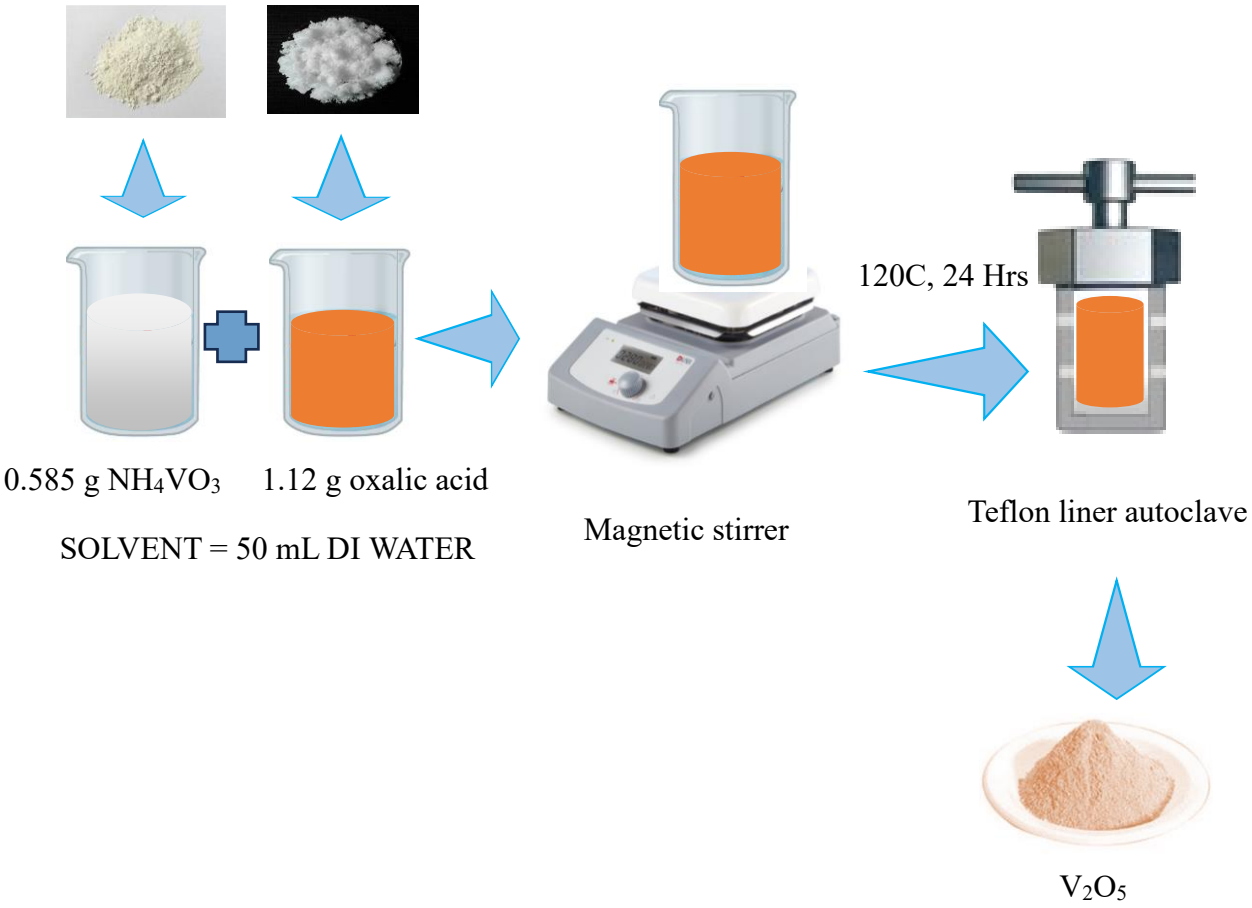

Fig. 3.5 Schematic representation of synthesis of V_2O_5.

At first, 585 mg NH_4VO_3 dissolved in 50 ml deionized water, and 1120 mg oxalic acid was added to the system to form an orange solution. The resulting solution was stirred for 30 minutes with the help of a magnetic bead. Then, concentrated HCl was added dropwise to the mixture and mixed well. The mixture was initially neutral, but the solution was allowed to achieve pH 3 on adding HCl. The solution was stirred for another 30 minutes to complete the reaction. The resulting solution was transferred to a Teflon-lined stainless-steel autoclave and heated in an oven at 120°C for 24 hours. It was then cooled down to room temperature and ppt was collected, centrifuged, and washed with distilled water 3-4

times, followed by washing with ethanol 2 times. Finally, the washed ppt was collected, transferred to a petri dish, and dried at 70 °C for 4-5 hours.

3.3.2 Solvothermal synthesis Ni@V_2O_5

The different percentage of Ni intercalation in V_2O_5 leads to the formation of different compounds.

a) **10% Ni intercalation over V_2O_5**: First, 150 mg of V_2O_5 (synthesized) was dissolved/dispersed in 60 mL ethanol. Then, 74.25 mg of $Ni(NO_3)_2 \cdot 6H_2O$ was added to the solution. The resulting mixture was stirred for 15 minutes and subsequently transferred into a Teflon-lined stainless-steel autoclave. It was then heated in an oven at 120 °C for 24 hours and allowed to cool naturally to room temperature. The resulting black precipitate was collected by centrifugation, washed several times with absolute ethanol, and dried at 70 °C for 3 hours to obtain the final composite, 10% Ni@V_2O_5.

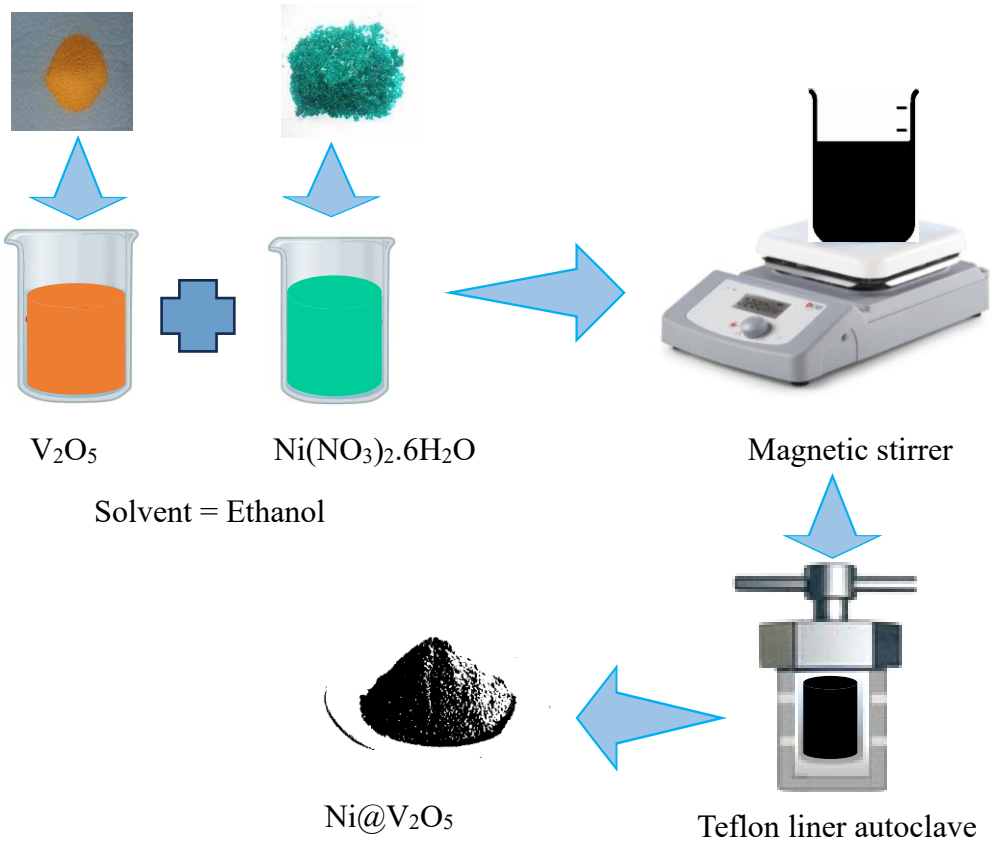

Fig. 3.6 Schematic representation of synthesis of Ni@V_2O_5

b) **20%, 30%, and 40% Ni intercalation over V_2O_5**: 20–40%Ni@V_2O_5 composites were synthesized by dispersing 150 mg of V_2O_5 in 60 mL of ethanol and adding the respective amounts of $Ni(NO_3)_2 \cdot 6H_2O$ (74.25, 148.50, 222.75, and 297.00 mg for 10%, 20%, 30%, and 40% Ni). The mixtures were stirred for 15 min, hydrothermally treated at 120 °C for 24 h, and the resulting black precipitates were collected, washed, and dried at 70 °C for 3 hr to get the final composites.

3.4 Fabrication of electrodes

Before surface modification, the Nickel foam was ultrasonically treated with dilute HCL, then with Millipore water and followed by acetone for 10 min each for the removal of surface impurities. This nickel foam electrode is used as a current collector. To produce different modified electrodes, 6 mg of synthesized nanocomposite materials was dispersed in 500 µl IPA and sufficient amount of Nafion(binder), which then placed in an ultrasonic bath for 20 minutes to get a suspension. This suspension was then drop-casted over a Nickel foam electrode (1cm ×1cm) and dried at 70°C for 2 hrs.

3.5 Electrochemical studies of the synthesized materials

The electrochemical experiments were conducted using a PARSTAT 4000 potentiostat, controlled through Ametek Versa Studio software. These studies were performed in a three-electrode configuration, with the working electrode being a modified nickel foam. The counter electrode was a platinum wire, and a reference electrode was an Ag/AgCl electrode, all immersed in a 2M potassium hydroxide (KOH) solution as the electrolyte. The specific capacitance of the synthesized materials was determined by substituting the appropriate values from the CV curves into Equations (3.2) and (3.3).

$$C_{sp} = \frac{\int IdV}{2\nu m \Delta V} \quad \text{.....(3.2)}$$

$$C_{sp} = \frac{A}{2\nu m \Delta V} \quad \text{..... (3.3)}$$

Where, m denotes active mass, the applied voltage is symbolized as ΔV, C_{sp} is the specific capacitance, and scan rate (ν), the area under the CV curve, in this case, is symbolized as A.

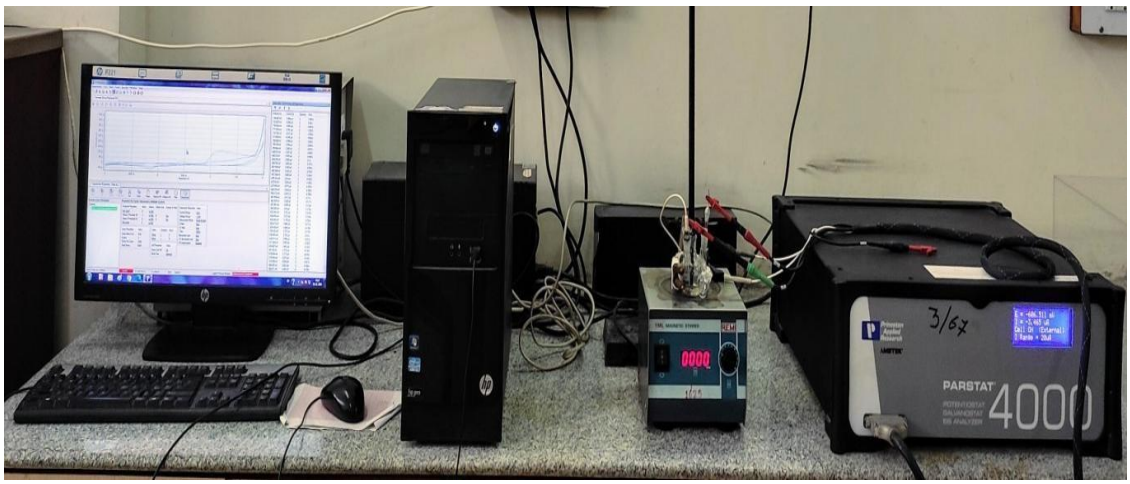

Fig. 3.7. Electrochemical setup

3.6 Scheme:

After successfully characterizing the synthesized Ni@V_2O_5, its applicability as supercapacitor material was investigated.

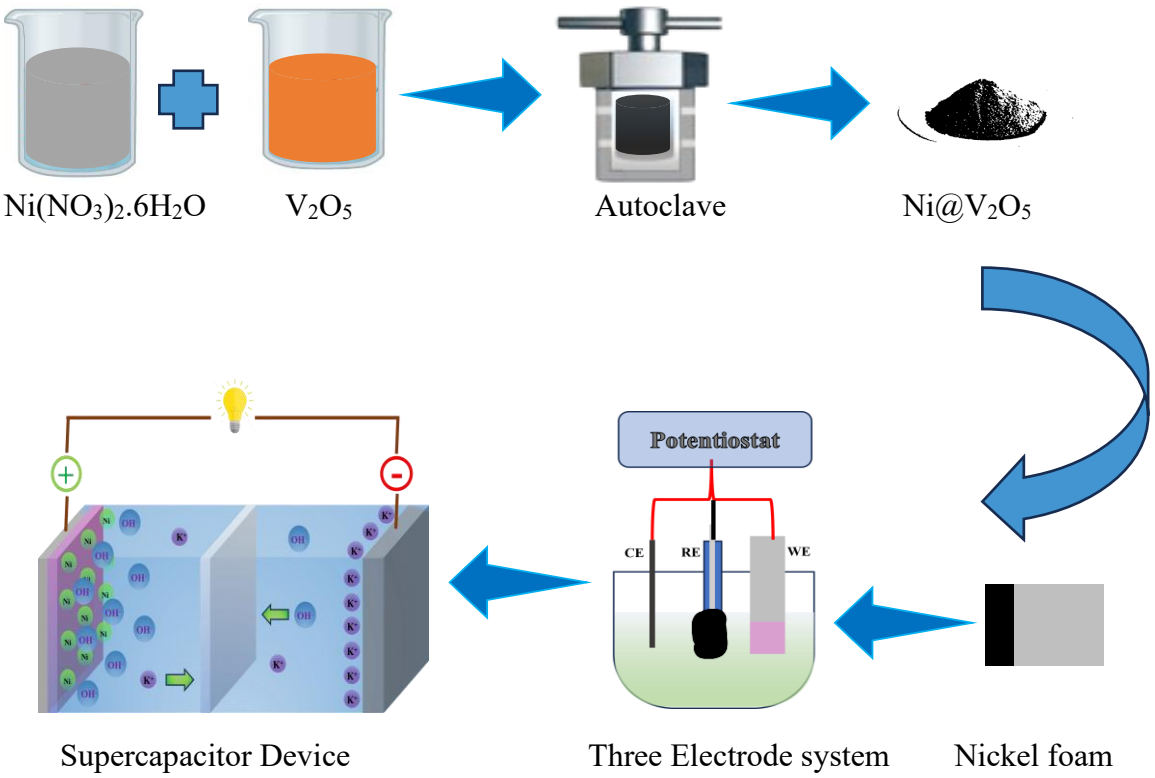

Fig. 3.8 Scheme representing use of synthesized materials in supercapacitor application

Starting with synthesis of V_2O_5, using ammonium metavanadate (NH_4VO_3) and oxalic acid and then using different percentages of nickel nitrate for various Ni- intercalated V_2O_5 to form Ni@V_2O_5. Synthesized 10%, 20%, 30%, and 40% Ni intercalated V_2O_5 and then characterized using techniques like FT-IR, XRD, FE-SEM, EDS, and TGA. Further it was drop-casted on the nickel foam and used as working electrode to check applicability in a supercapacitor device.

Chapter 4
Result and Discussion

4.1 Characterization of Synthesized Materials

4.1.1 FTIR

The FTIR spectra for the synthesized V_2O_5 and its nano-composite are given below.

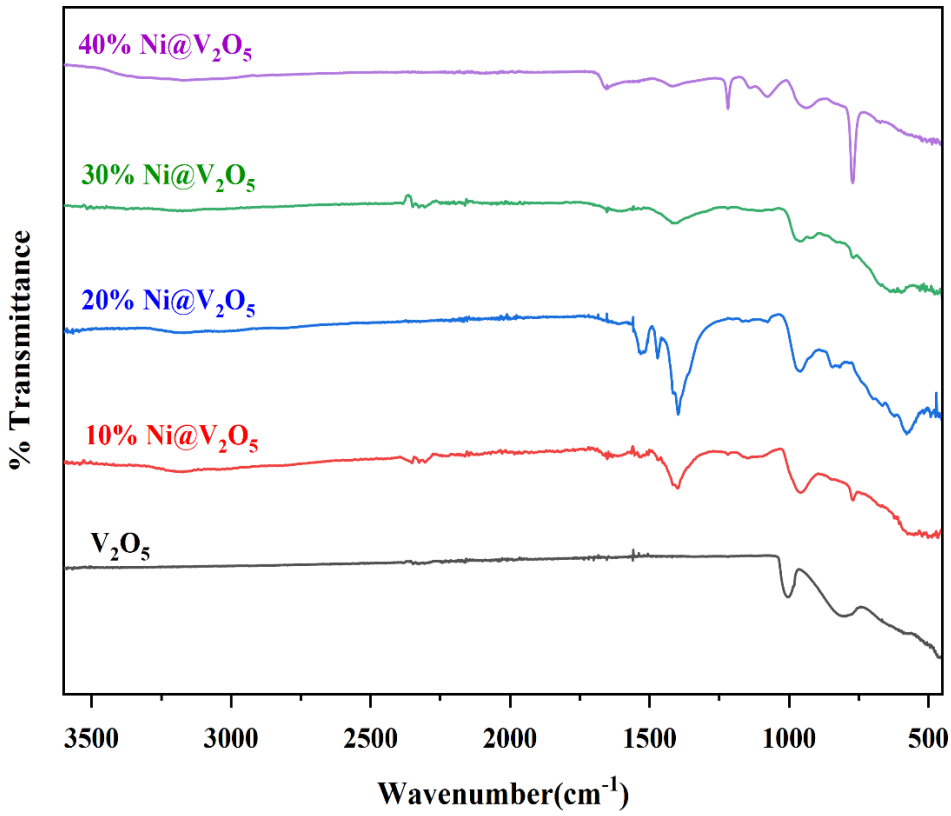

Fig.4.1 FTIR spectra of V_2O_5 and its different % Ni intercalated V_2O_5 nanocomposite.

In Fig.4.1 the FT-IR spectra of the V_2O_5 and 10%, 20%, 30%, and 40% Ni intercalated V_2O_5, the common peak in all the above spectra around 1000 cm^{-1} is due to the presence of V-O stretching vibration, along with V-O-V stretching vibrations around 800 cm^{-1}. This conforms the V-O bond present in the synthesized compounds.

4.1.2 XRD

The XRD diffractogram for the synthesized V_2O_5 and its nano-composite is given below.

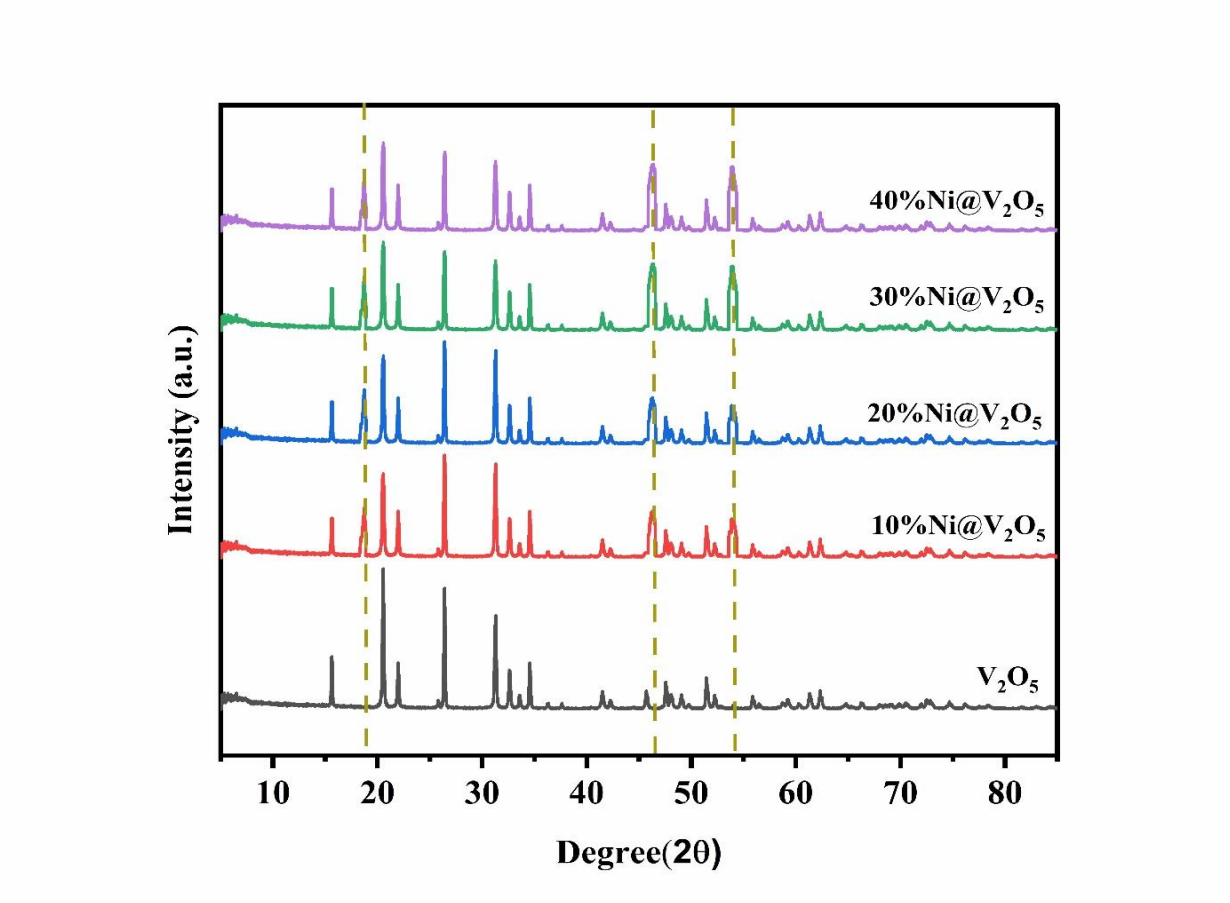

Fig. 4.2 XRD diffractogram of V_2O_5 and different percent Ni-intercalated V_2O_5 nanocomposite

Figure 4.2 shows XRD diffractogram of V_2O_5, 10%, 20%, 30%, and 40% Ni-intercalated V_2O_5 nanocomposite. Sharp and highly intense Bragg's diffraction peaks in XRD reveals that the synthesized material is highly crystalline and matches with the JCPDS Card no. (41-1426). The extra intense peak denoted by violet dotted lines corresponds to Ni, which is absent in XRD spectra of V_2O_5 but present in 10%, 20%, 30%, and 40% Ni intercalated V_2O_5 nanocomposites, validating the successful intercalation of Ni in V_2O_5 without disturbing the planes of pristine V_2O_5. This shows the successful synthesis of our Ni intercalated V_2O_5 nanocomposites.

4.1.3 FE-SEM and EDX:

The SEM image for the synthesized V_2O_5 and its nano-composite is given below.

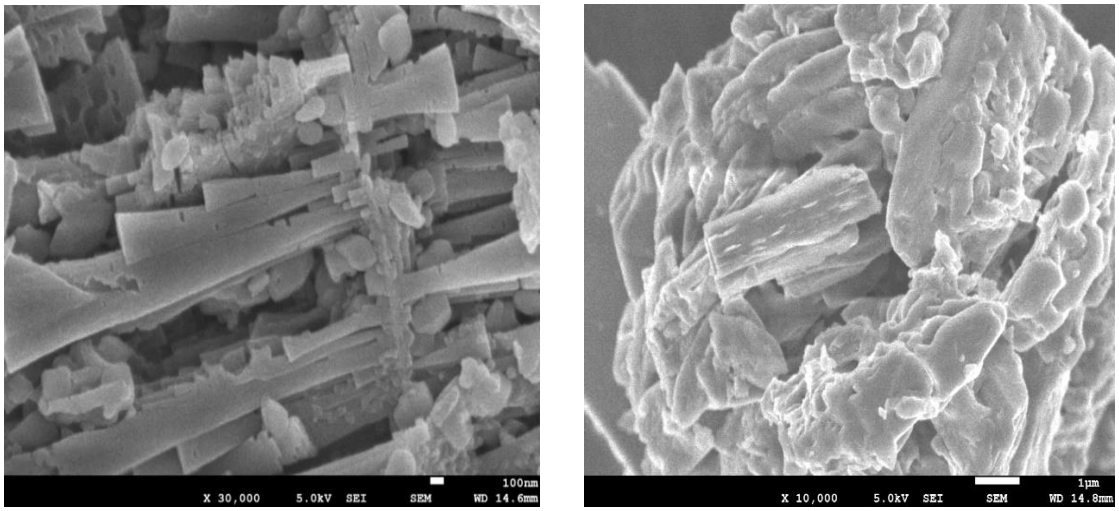

Fig. 4.3 (a) FE-SEM of synthesized V_2O_5.

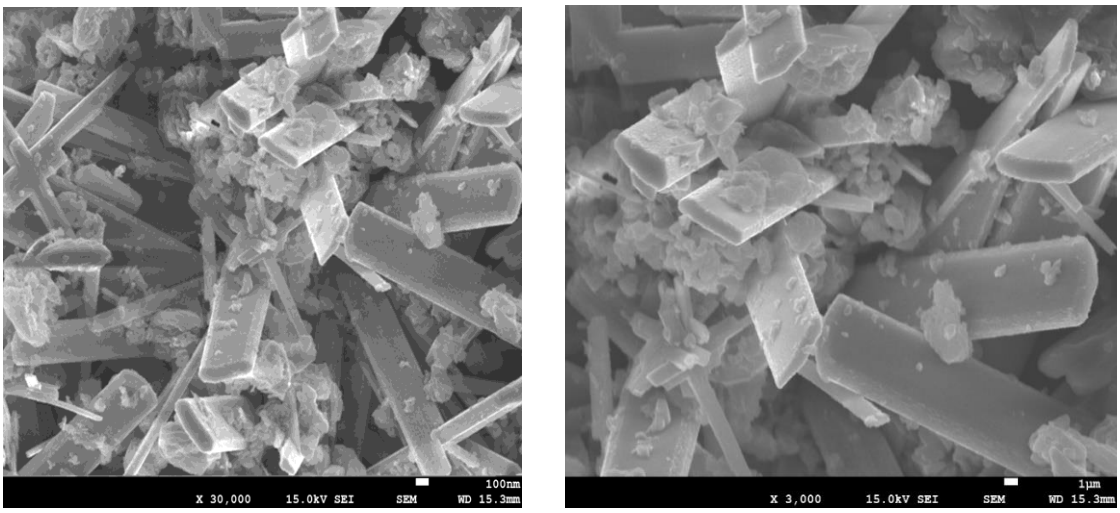

Fig. 4.3 (b) FE-SEM of synthesized 20% Ni intercalated V_2O_5.

The surface morphology and microstructural features of the synthesized V_2O_5 and 20% Ni@V_2O_5 composite were investigated using Field Emission Scanning Electron Microscopy (FE-SEM). As shown in Fig. 4.3 (a), the FE-SEM images of pristine V_2O_5 reveal the formation of well-defined elongated nanorod-like structures. The rod-like morphology is characteristic of crystalline V_2O_5 synthesized via solvothermal methods and is known to contribute to improved surface area and electron transport properties in various

applications. In contrast, the FE-SEM image of the 20% Ni@V_2O_5 composite displays a similar nanorod-like morphology with additional surface modifications (Fig. 4.4 (b)). The presence of nickel is evident from the fine nanoparticles that appear to be intercalated or anchored onto the surface of the V_2O_5 nanorods. These Ni particles are distributed uniformly over the rods, indicating effective dispersion and strong interaction between the Ni species and the V_2O_5 matrix.

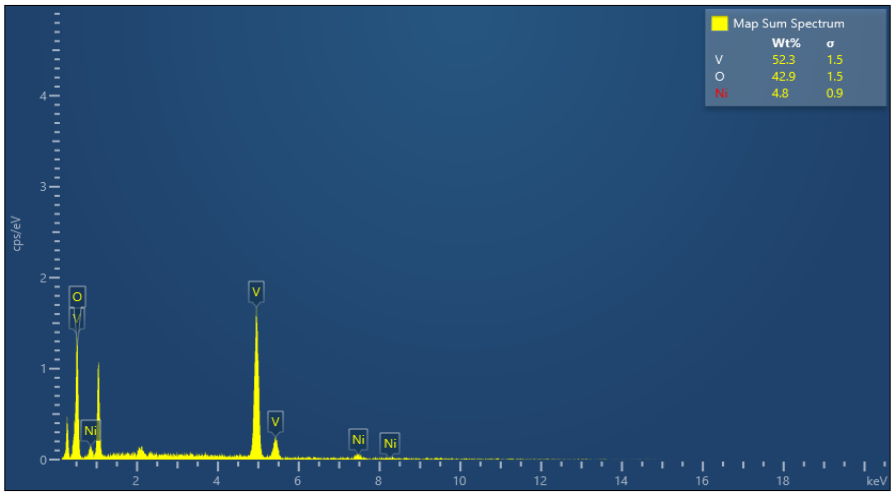

Fig. 4.4. EDS spectra of 20% Ni@ V_2O_5

Fig.4.4 shows the elemental composition of 20% Ni@ V_2O_5, which was carried out by the Energy Dispersive X-ray spectroscopy (EDS). The EDS spectra reveal that V, O and Ni are the major elements present in the nanocomposite. There is a small peak corresponding to the Ni which inferring the fact that it is the intercalation element. Fig.4.5 Corresponds to the elemental mapping imaging of Vanadium (V), Nickel (Ni) and Oxygen (O).

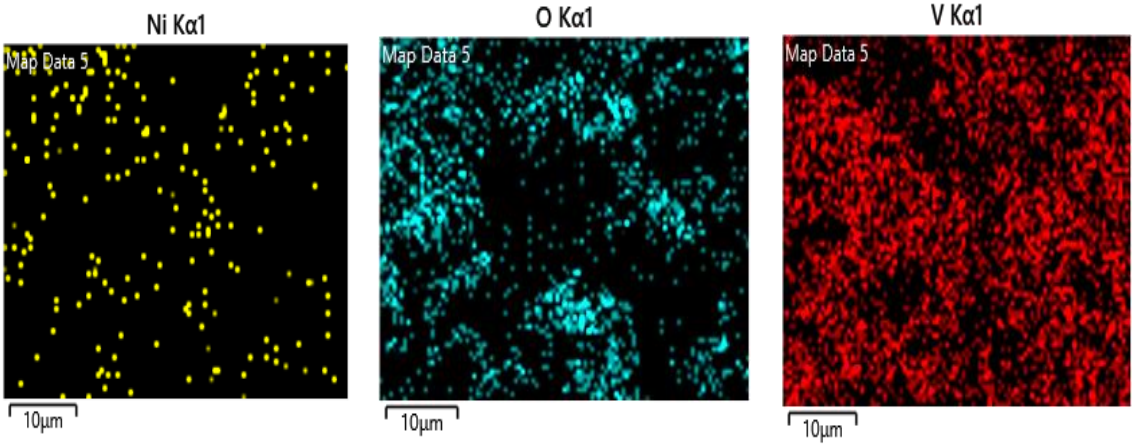

Fig.4.5 Elemental mapping of 20% Ni@ V_2O_5

4.1.4 TGA (Thermogravimetric analysis)

Thermogravimetric Analysis, which is a technique used to measure the change in the mass of a sample as it is heated or cooled over time. The primary purpose of TGA is to analyze the thermal stability of the materials with respect to the change in temperature.

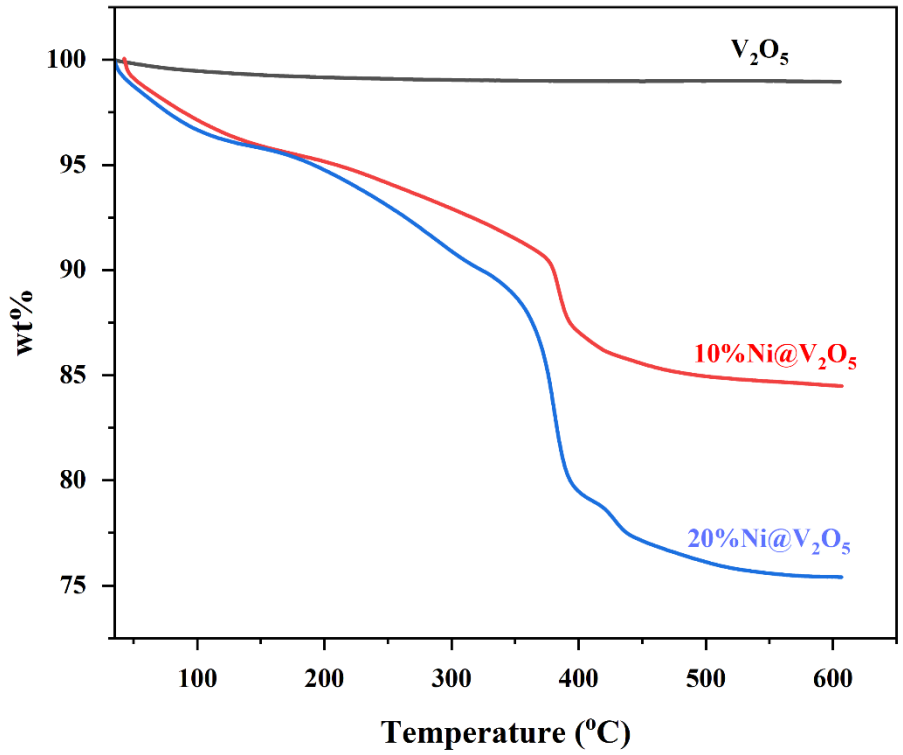

Fig.4.6 TGA spectrum for V_2O_5 and its Ni intercalated nanocomposite

In Figure 4.6 TGA spectrum of the V_2O_5 sample shows a minimum loss of weight of the sample, approximately 1.5% initially, in between 35°C to 200°C due to the removal of moisture or solvent from the sample. From 200-800°C, there is no significant change in the mass of V_2O_5, revealing its excellent thermal stability. 10% and 20% Ni intercalated V_2O_5 sample shows 15% and 25% of initial weight loss, between 35°C to 200°C, due to the removal of moisture or solvent from the sample. Then, after a gradual decrease between 300 to 450°C occurs due to the removal of impurities bonded with nickel and its conversion to Ni oxide. Above 450°C, both composites show a constant phase, which corresponds to the thermal stability of the V_2O_5.

4.2 Supercapacitor study

4.2.1. Cyclic Voltammetry:

To evaluate the electrochemical performance of pristine V_2O_5 and Ni-intercalated V_2O_5 materials, cyclic voltammetry (CV) measurements were conducted in a three-electrode configuration using 2 M KOH aqueous electrolyte. The CV curves were recorded in a potential window ranging from –0.1 V to 0.5 V vs. Ag/AgCl at a scan rate of 1 mV/s. Ni was intercalated into the V_2O_5 matrix at varying concentrations of 10%, 20%, 30%, and 40% by weight to investigate its effect on specific capacitance.

Among all the synthesized samples, the 20% Ni-intercalated V_2O_5 (denoted as 20% Ni@V_2O_5) exhibited the highest specific capacitance, reaching a remarkable value of **1256.25 F g^{-1}** at 1 mV s^{-1}. This superior capacitive performance of 20%Ni@V_2O_5 because 20% Ni loading provides an optimal balance between increased electrical conductivity and the preservation of the layered V_2O_5 structure. Ni ions likely intercalate into the van der Waals gaps of the V_2O_5 layers, facilitating faster electron and ion transport. The presence of Ni species enhances faradaic redox reactions due to the multiple oxidation states of Ni (Ni^{2+}/Ni^{3+}), which contribute additional Pseudocapacitance beyond the intrinsic redox activity of V_2O_5. The moderate Ni intercalation reduce charge transfer resistance, resulting in better reversibility and faster ion diffusion at the electrode/electrolyte interface.

In contrast, both lower (10%) and higher (30% and 40%) Ni contents exhibited comparatively lower specific capacitances. At lower concentrations, the improvement in electrical conductivity and redox activity is insufficient, while at higher Ni loadings, excessive intercalation may distort the V_2O_5 lattice, block ion diffusion channels, or lead to agglomeration, thereby hindering electrochemical performance.

These results highlight that a 20% Ni-intercalation into V_2O_5 provides the optimal structural and electrochemical synergy required for high-performance supercapacitor electrodes.

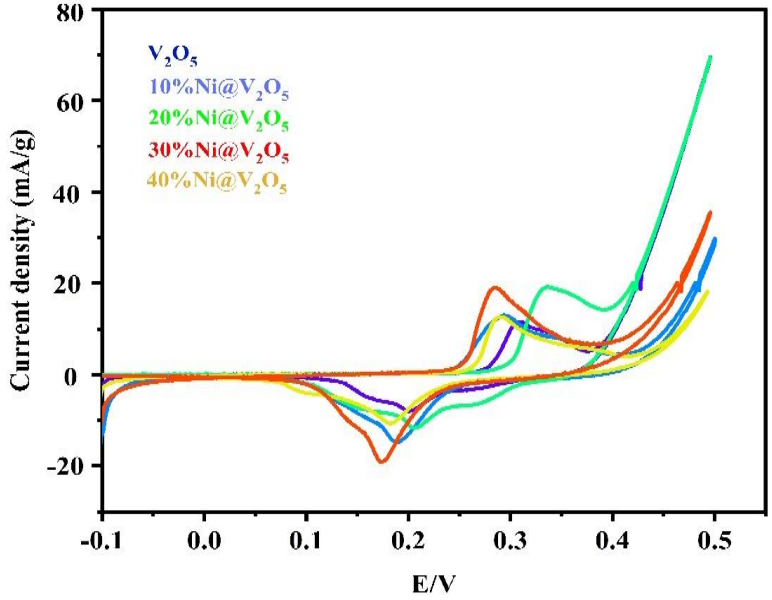

Fig.4.2.1 Cyclic voltammograms of different electrode materials at 1 mV/s scan rate

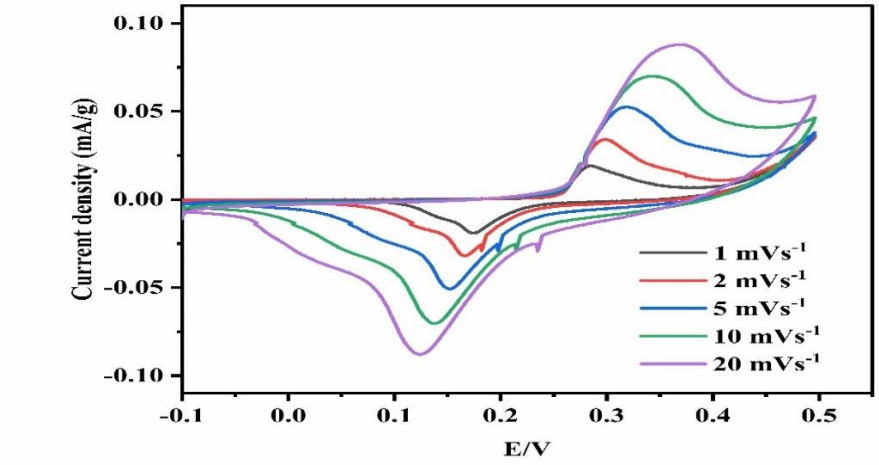

Fig.4.2.2 Cyclic voltammogram curve of V_2O_5 at different scan rate

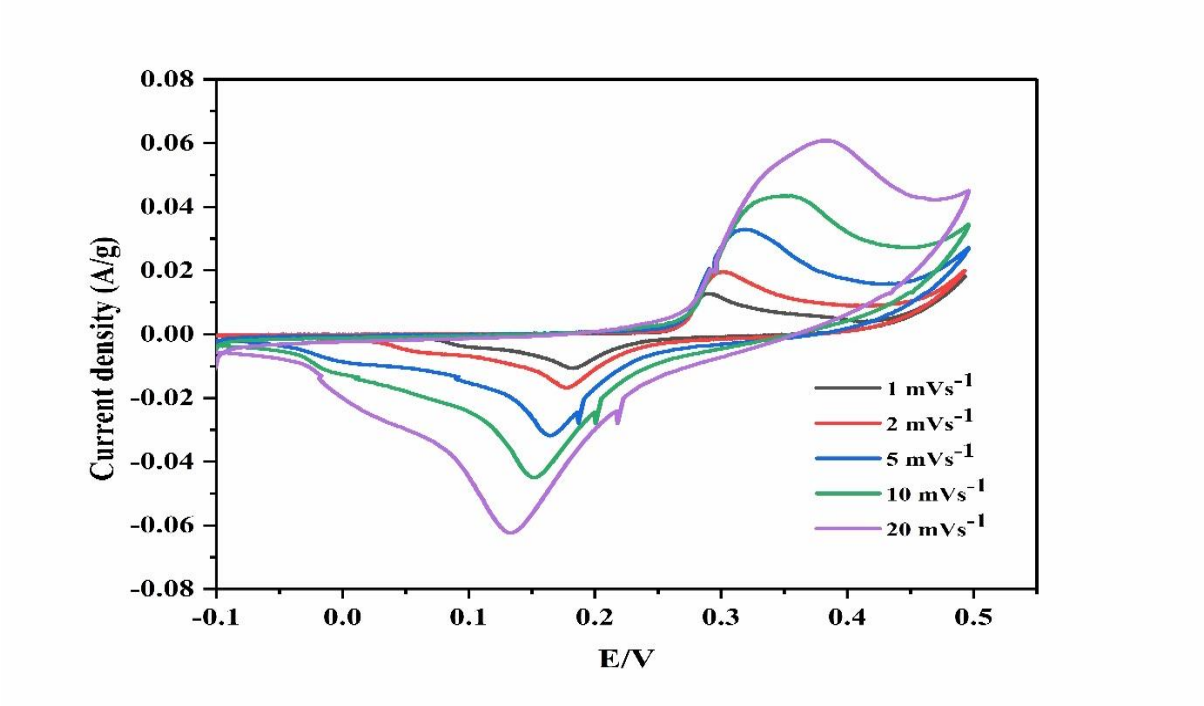

Fig.4.2.3 Cyclic voltammogram curve of 20% Ni@ V_2O_5 at different scan rates

4.2.2 Galvanostatic charge discharge (GCD)

To further investigate the electrochemical performance of pristine and Ni-intercalated V_2O_5 materials, Galvanostatic Charge–Discharge (GCD) tests were carried out in a three-electrode configuration using 2 M KOH aqueous electrolyte. The measurements were performed within a potential window of –0.1 V to 0.5 V at a constant current density of 5 A g^{-1} (Fig. 4.3.1). Among all the tested samples, the 20% Ni-intercalated V_2O_5 (20% Ni@V_2O_5) electrode demonstrated the highest specific capacitance of 1518 F g^{-1} at a current density of 5 A g^{-1}. This exceptional performance may be due to, at the 20% level, the intercalated Ni ions can improve electron transport pathways without significantly distorting the layered structure of V_2O_5. This promotes efficient ion diffusion and better electrochemical reversibility, as reflected in the nearly symmetric charge–discharge curves. Unlike higher Ni loadings (30% and 40%), which may lead to particle agglomeration and block active sites, the 20% intercalation maintains good dispersion of Ni species and preserves the porous morphology of the electrode material. In contrast, lower (10%) and higher (30% and 40%) (**Fig.4.3.2 shows Csp of V_2O_5 at different current densities**) Ni contents yielded relatively reduced specific capacitance values, suggesting suboptimal redox activity and structural effects at those levels. Similarly, the

Specific capacitance of V_2O_5 and 20%Ni@V_2O_5 was calculated at various current densities i.e., from 5-20 A/g which shows a decrease in the specific capacitance shown in **Fig.4.3.3** (Table 4.4).

Overall, the GCD analysis corroborates the CV results and confirms that 20% Ni intercalation into V_2O_5 provides the most favorable configuration for achieving high specific capacitance and robust charge storage performance in alkaline supercapacitor systems.

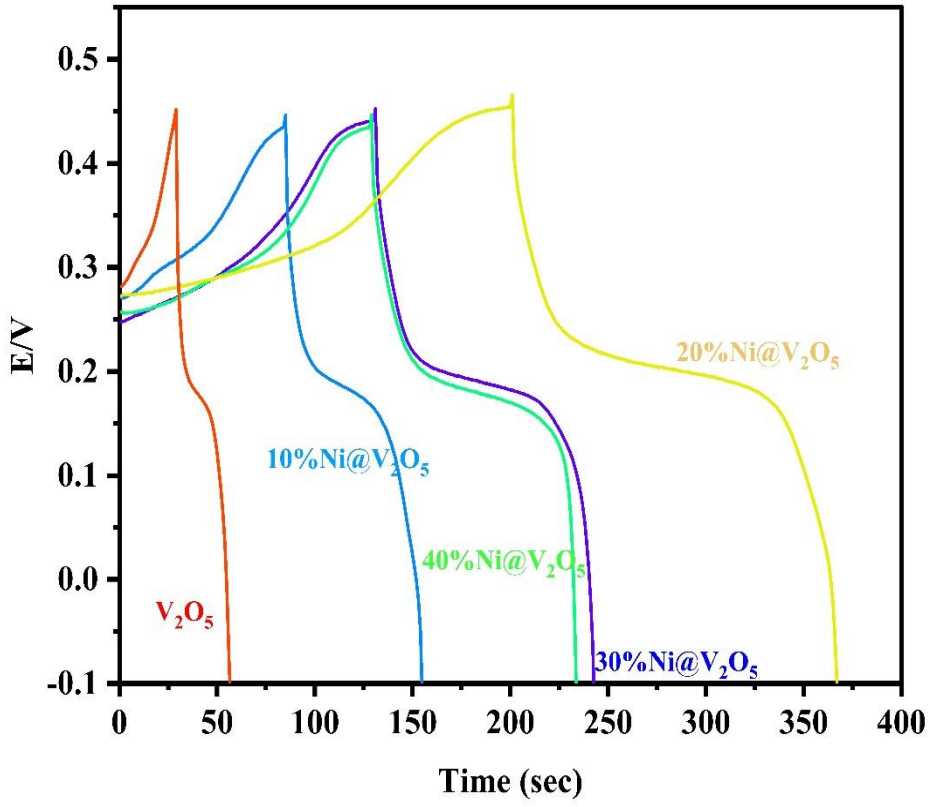

Fig.4.3.1 GCD plots of different electrode materials at 5 A/g current densities

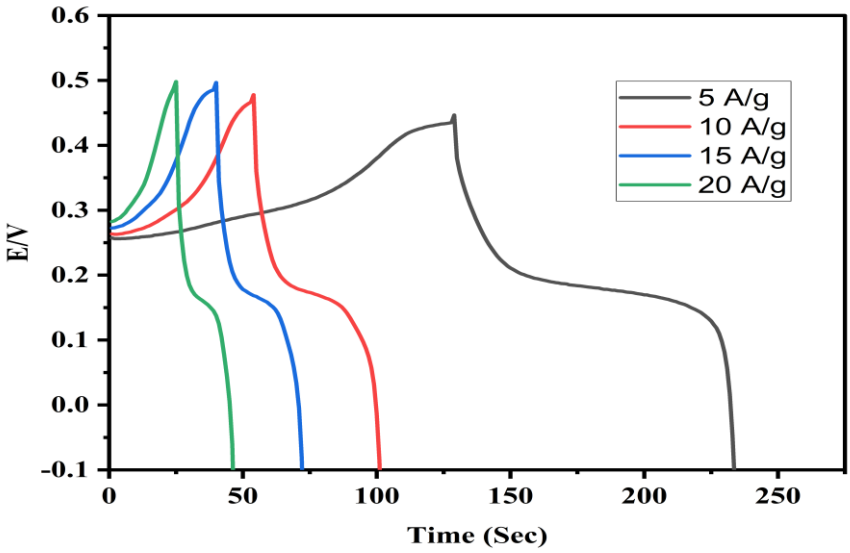

Fig.4.3.2 GCD plots of V_2O_5 electrode materials at different current densities

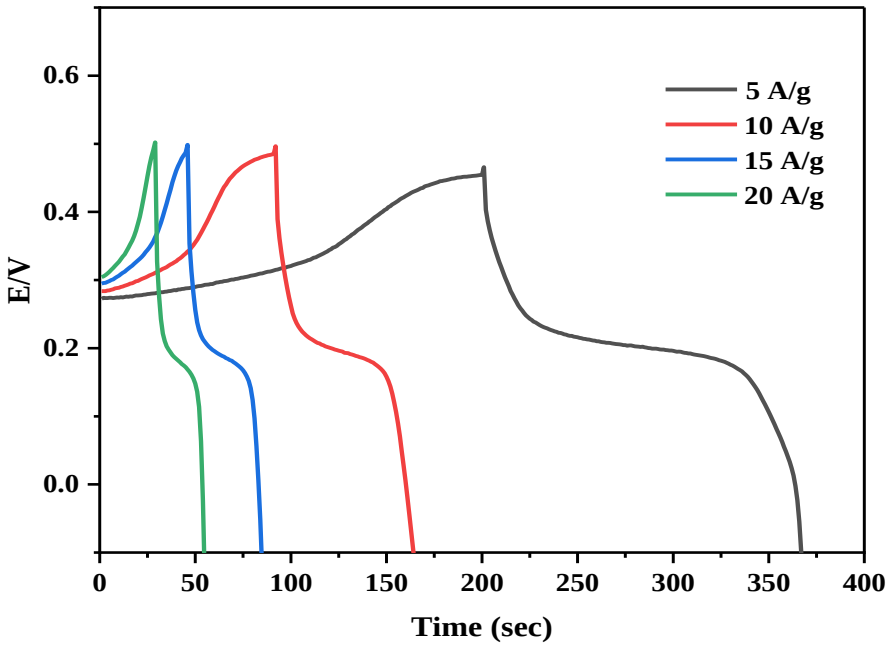

Fig.4.3.3 GCD plots of 20% Ni@V_2O_5 electrode materials at different current densities

Specific Capacitance value as a function of area under the curve of CV and GCD are given Table 4.1) different electrodes for at scan rate of 1 mV/s, Table 4.2) Csp of 20%Ni@V_2O_5 at different scan rates, Table 4.3) Csp of different electrodes for at 5 A/g current density, Table 4.4) Csp of 20%Ni@V_2O_5 at different current densities.

Table 4.1

S. No.	Electrode materials	Specific capacitance (F/g)
1	V_2O_5	769.16
2	10% NiV_2O_5	927.50
3	20% NiV_2O_5	1294.25
4	30% NiV_2O_5	1094.58
5	40% NiV_2O_5	859.16

Table 4.2

Sr. No.	Scan rate (mV/s)	Specific Capacitance (F/g)
1	1	1256.25
2	2	892.08
3	5	702.33
4	10	524.70
5	20	383.04

Table 4.3

S. No.	Electrode materials	Specific capacitance (F/g)
1	V_2O_5	640.90
2	10% NiV_2O_5	1026.45
3	20% NiV_2O_5	1518
4	30% NiV_2O_5	963.36
5	40% NiV_2O_5	820.90

Table 4.4

Sr. No.	Current density (A/g)	Specific Capacitance (F/g)
1	5	1518
2	10	1311.45
3	15	905.91
4	20	830.33

Chapter 5

Conclusion and Future Scope

5.1 Conclusion

In this study, the structural and morphological analysis of pristine and Ni-intercalated V_2O_5 materials was systematically evaluated using FT-IR, XRD, FE-SEM, EDS and Elemental mapping which confirms its successful synthesis. Electrochemical performance was evaluated using cyclic voltammetry (CV) and galvanostatic charge–discharge (GCD) techniques in a three-electrode setup with 2 M KOH as the electrolyte. Among the various compositions investigated, the 20% Ni-intercalated V_2O_5 (20% Ni@V_2O_5) demonstrated the highest specific capacitance, reaching 1256.25 F g^{-1} at 1 mV s^{-1} (CV) and 1518 F g^{-1} at 5 A g^{-1} (GCD). This superior performance is attributed to the optimal balance between enhanced electrical conductivity and preservation of the V_2O_5 layered structure achieved at this Ni concentration. The intercalated Ni ions not only improved charge transport and facilitated fast redox reactions due to their multiple oxidation states but also reduced charge transfer resistance, enhancing electrode/electrolyte interfacial kinetics. Lower (10%) and higher (30% and 40%) Ni loadings resulted in suboptimal performance due to insufficient conductivity enhancement or structural distortion and ion transport hindrance, respectively. The study confirms that 20% Ni intercalation is the most effective configuration for maximizing pseudocapacitive behavior in alkaline supercapacitor applications.

5.2 Future scope

Building upon the promising results exhibited by 20% Ni@V_2O_5 electrode, future research could be focused on the following areas:

1. **Long-Term Cycling Stability:** Investigate the cycling durability and coulombic efficiency over extended charge–discharge cycles to assess the practical viability of the material in real-world supercapacitor devices.
2. **Full-Cell Assembly:** Fabricate asymmetric or symmetric two-electrode supercapacitor devices using 20% Ni@V_2O_5 as the positive electrode to evaluate energy and power densities under practical conditions.

3. **Electrolyte Optimization:** Explore the performance of 20% Ni@V_2O_5 in alternative aqueous or gel-based electrolytes (e.g., Na_2SO_4, PVA–KOH) to improve safety and electrochemical window.
4. **Hybrid Composites:** Combine Ni@V_2O_5 with conductive materials like graphene, carbon nanotubes, or MXenes to form hybrid electrodes with synergistic effects for improved rate capability and mechanical stability.
5. **Scalability and Cost Analysis:** Study the synthesis scalability and economic feasibility of Ni intercalation methods for potential commercial application in energy storage devices.

Chapter 6
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